ELSEVIER

Journal of Nuclear Materials 254 (1998) 74–77

journal of nuclear materials

Letter to the Editors

Deuteron irradiation creep of chemically vapor deposited silicon carbide fibers

R. Scholz *

CEC Joint Research Centre Ispra, T.P. 202, 21020 Ispra (Va), Italy

Received 29 September 1997; accepted 28 November 1997

Abstract

Irradiation creep tests were conducted on Textron SCS-6 silicon carbide (SiC) fibers during irradiation with 14 MeV deuterons at 450 and 600°C. The fibers are produced by a CVD procedure; their microstructure may therefore be representative for the matrix of a SiC composite. There is a significant radiation induced increase in creep deformation. Both quantities, irradiation creep strain and creep rate, are higher at 450°C than at 600°C for doses <0.07 dpa. © 1998 Elsevier Science B.V.

1. Introduction

Silicon carbide composites are considered as structural materials for the first wall and blanket of nuclear fusion reactors [1,2]. This material is attractive mainly because of its low activation under neutron irradiation, which is beneficial for reactor maintenance and waste disposal. These two aspects are important for availability and public acceptance of future fusion reactors.

The effects of high-energy particle irradiation on the mechanical properties of SiC, SiC-fibers or SiC-composites have been determined predominantly in post-irradiation tests [2]. Light ion irradiation creep experiments conducted on SiC in the temperature range between 235 and 505°C were not conclusive [3]. The observed length increase of the specimens showed a less than linear stress dependence and was attributed to swelling of the material although a creep compliance $\kappa = 5 \times 10^{-10}$ Pa^{-1} dpa^{-1} similar to that of UO_2 at 400°C could not be excluded [3]. ($\kappa = (\mathrm{d}\varepsilon/\mathrm{d}t)/K\sigma$, $\mathrm{d}\varepsilon/\mathrm{d}t$ is the tensile strain rate, K is the damage rate and σ is the applied tensile stress.) In the temperature range below 1000°C, swelling of SiC occurs without an incubation dose and saturates rapidly at doses smaller than 0.5 dpa. The linear expansion observed after saturation declined from about 1% at room temperature to 0.05% at 1000°C [4]. So, the magnitude of swelling is sufficiently high to mask the irradiation creep strain in a tension creep test where the swelling induced linear expansion adds directly to the total strain of the specimen. The opposite effect occurs in torsion tests on cylindrical specimens: the shear strain γ decreases if the specimen volume increases. However, this decrease in γ is small in comparison to the expected creep strain if standard values for swelling and creep rate of SiC are assumed, and the effect of swelling on the creep strain may be neglected.

2. Specimens

Light ion irradiation tests require the use of mini specimens for damage homogeneity and cooling reasons. The available particle energy and the capacity of the cooling system limit the specimen dimensions. For the Ispra cyclotron conditions, the thickness of the irradiated SiC specimen should not exceed 200 μm, if the inhomogeneity in damage has to stay below 15–20%. The commercially available TEXTRON SCS-6 fiber having a diameter of 142 μm fulfills this condition and has been used for the irradiation creep tests. A detailed description of composition and microstructure of the SCS-6 fiber is given in [5] mainly based on studies carried out by Ning and

* Tel.: +39-332 789 060; fax: +39-332 785 388.

PII S0022-3115(97)00358-9

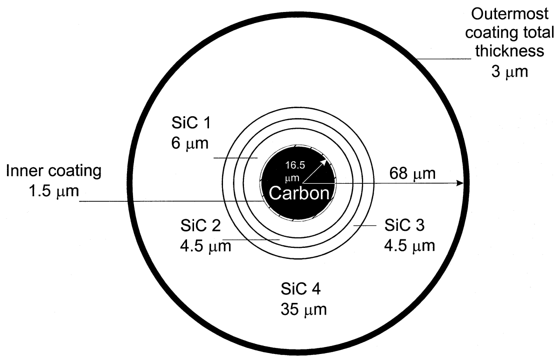

Fig. 1. Concentric layer structure of the TEXTRON SCS-6 fiber (from Ning and Pirouz [7]).

co-workers [6,7] which showed that the fiber consists of a number of concentric layers (see Fig. 1): the carbon core of 33 μm diameter is covered by a thin graphite layer on which a SiC mantle of 50 μm thickness is deposited by applying a chemical vapor deposition procedure (CVD) followed by the outside carbonaceous coating of 3 μm thickness. The SiC sheath of the fiber has four sublayers, which consist of β-SiC crystallites of columnar shape with the long axis preferentially aligned in radial direction of the fiber. The transition from one layer to the next is characterized by an increase in size of the crystallites and the degree of their orientation. The crystallite length amounts to 5–15 nm close to the carbon core and reaches about 10–12 μm in layer four. Layer four contains almost perfectly stoichiometric β-SiC while layers 1, 2 and 3 have 10–20% excess carbon.

The layer structure of the fiber affects tests in torsion, since the shear stress γ is not constant throughout the specimen volume, but is zero at the specimen axis and has its maximum value at the outer specimen surface. In a torsion creep test on cylindrical specimens of length L and radius R, the twist angle ϕ is measured as a function of time and the shear strain $\gamma = \phi r/L$ is calculated, where r is the distance from the specimen centerline. For a homogeneous specimen, $\phi = 2ML/(\pi G R^4)$ within the elastic range, to which the applied stresses are limited (G is the shear modulus, M stands for the applied torque). Fatigue tests in torsion showed that the fiber contains two weak interfaces where debonding occurred [8]: the inside coating close to the carbon core and the outside coating. For this reason, the maximum shear stress $\tau = 2M/\pi R_1^3$, and shear strain $\gamma = \phi R_1/L$ are calculated with the relationship for a hollow tube like specimen of outer radius R_1 and inner radius R_2: $\phi = 2ML/[(G(R_1^4 - R_2^4)]$ by adopting the values for the inside radius R_2 and the outside radius R_1 of the SiC sheath.

The SCS-6 fibers are produced by a CVD procedure. The resulting polycrystalline silicon carbide is basically the same material as polycrystalline SiC produced by chemical vapor infiltration that is the process most commonly used to fabricate SiC matrix composites [9]. So, the irradiation creep behavior of the SCS-6 fiber is likely to be similar to that of a SiC composite matrix.

3. Results

The results are gathered in Figs. 2 and 3. Fig. 2 shows the strain time behavior of a specimen subjected to thermal

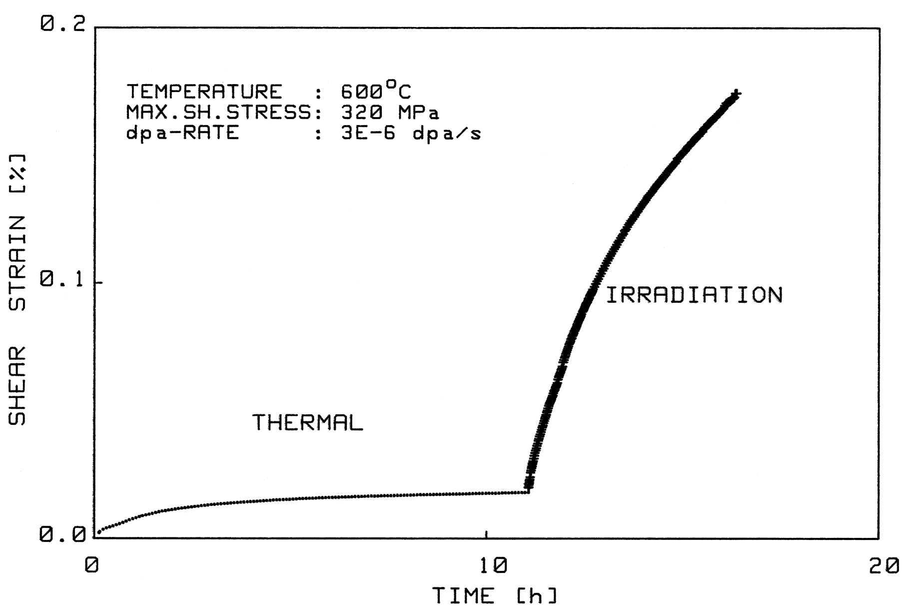

Fig. 2. Creep curve for thermal and irradiation conditions.

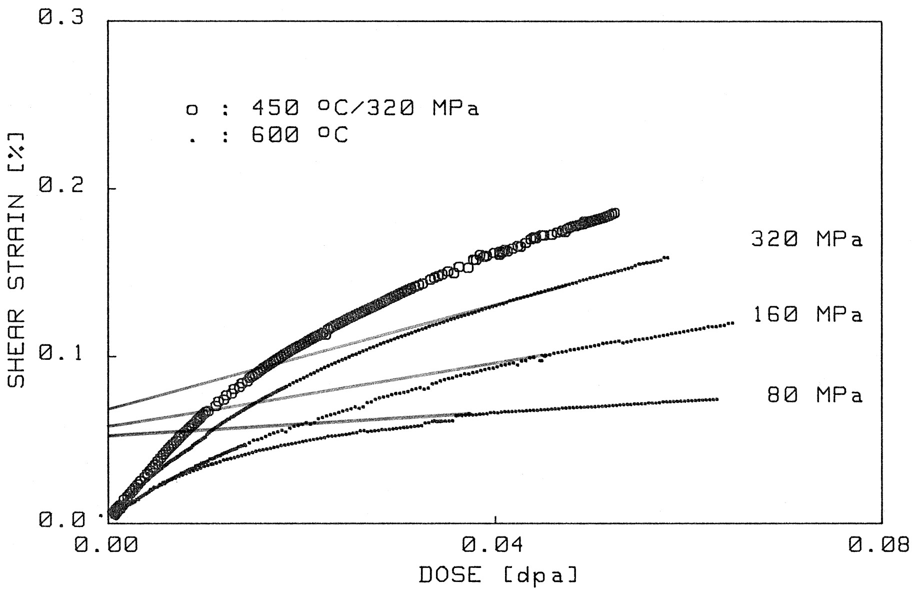

Fig. 3. Shear strain as a function of the accumulated dose. The slope of the straight lines is equal to the steady state creep rate. The magnitude of the transient creep strain is given by the intersection of the extrapolated line and the ordinate.

and irradiation conditions at 600°C and a maximum shear stress of 320 MPa. During the thermal creep period, the shear strain reaches saturation after ca. 12 h and the creep rate approximates zero. There is a drastic increase in strain as soon as the specimen is irradiated. The creep rate, $s = d\gamma/dt$, decreases during the irradiation phase, the drop in creep rate is strong at the beginning of the irradiation and slows down with time. When the irradiation is switched off still maintaining stress and temperature at the same values, the strain rate becomes immediately zero.

Fig. 3 shows the creep curves for different stresses at 600°C and one curve for 450°C, the shear strain is plotted versus dose. For all curves measured, the creep rate slows down with the accumulated dose before reaching, after ~ 0.04 dpa, an approximately constant value s_s. In an attempt to rationalize the results, the total irradiation creep strain γ may be considered as the sum of two components, a transient strain component γ_t and a steady state component γ_s. The slope of the straight lines drawn in Fig. 3, is equal to s_s, the magnitude of γ_t is given by the intersection of the extrapolated line and the ordinate. Fig. 3 shows that the amount of transient irradiation creep strain varies only slightly with the applied maximum shear stress τ, while s_s increases linearly with τ.

Fig. 3 shows further that the curve measured for 450°C MPa lies above the 600°C curve, the applied shear stress being equal for the two tests. So, both quantities, creep strain and creep rate, are higher for 450°C over the whole dose range imposed indicating that the irradiation creep deformation drops if the temperature is increased. More tests are needed to confirm this relationship. However, a similar temperature dependence has been observed for the amount of swelling after neutron irradiation in the same temperature interval and a correlation of both effects, swelling and irradiation creep, is likely. TEM studies of β-SiC after neutron irradiation indicated that swelling, at temperatures below 1000°C, is caused by immobile point defects and/or small interstitial loops [10]. For a specimen under stress, the loops might nucleate preferentially on lattice planes with a component orthogonal to the applied stress direction. The stress induced preferential nucleation of loops (SIPN) gives a creep rate that can be correlated to the volume swelling rate [11].

At 450°C, irradiation creep has been measured for AISI 316L stainless steel with the same experimental set-up under deuteron irradiation [12] and a comparison between the two materials is possible by calculating the creep compliance κ defined as irradiation creep rate divided by damage rate and applied stress. For torsion test, κ is calculated with the tensile equivalents σ and ε for the torsional quantities, τ and γ, using the relationship $\varepsilon/\sigma = \gamma/3\tau$. At 450°C, the resulting creep compliance $\kappa = 2.1 \times 10^{-11}$ Pa^{-1} dpa^{-1} is equal for both materials, the SCS-6 fiber and 20% cold-worked AISI 316L stainless steel within the error limits.

4. Conclusions

Creep tests have been conducted in torsion on the TEXTRON SCS-6 silicon carbide fiber during irradiation with 14 MeV deuterons at 450 and 600°C up to a total dose of 0.06 dpa. The SCS-6 fibers are produced by a CVD procedure and may be considered as representative for the SiC matrix of a CVI produced SiC composite. The tests show that:

(1) The irradiation creep curves, for the dose range < 0.07 dpa, are characterized by a decreasing creep rate. The drop in creep rate slows down with the accumulated dose and reaches, after ~ 0.04 dpa, an almost constant value which is a linear function of stress at 600°C.

(2) Both quantities, irradiation creep strain and rate, are higher at 450°C than at 600°C.

(3) The irradiation creep compliance $\kappa = 2.1 \times 10^{-11}$ Pa^{-1} dpa^{-1} determined at 450°C is equal to that of 20% cold-worked stainless steel at the same temperature.

References

[1] L.L. Snead, Fusion Techn. 24 (1992) 65.

[2] R.H. Jones, D. Steiner, H.L. Heinisch, G.A. Newsome, H.M. Kerch, J. Nucl. Mater. 245 (1997) 87.

[3] Z. Zhu, P. Jung, J. Nucl. Mater. 212–215 (1993) 1082.

[4] R.J. Price, Nucl. Technol. 35 (1977) 320.

[5] A. Parvizi-Majidi, Fibers and Whiskers, in: Material Science and Technology, Vol. 13, VHC Verlag, 1993, pp. 27–83.

[6] X.J. Ning, P. Pirouz, K.P.D. Lagerlof, J. DiCarlo, J. Mater. Res. 5 (1990) 2865.

[7] X.J. Ning, P. Pirouz, J. Mater. Res. 6 (1991) 2234.

[8] R. Scholz, A. Frias Rebelo, F. dos Santos Marques, in: Proc. IEA Int. Workshop on SiC/SiC Ceramic Composites for Fusion Structural Applications, Oct. 28–29, 1996, Ispra (Va) Italy, p. 147.

[9] L.U. Ogbuji, T.E. Mitchell, A.H. Heuer, J. Am. Ceram. Soc. 4 (1981) 100.

[10] T. Yano, T. Iseki, Philos. Mag. A 62 (1990) K67.

[11] A.D. Brailsford, R. Bullogh, Philos. Mag. 27 (1973) 49.

[12] R. Scholz, J. Nucl. Mater. 212–215 (1994) 530.